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AN AUTORADIOGRAPHIC TECHNIQUE FOR STUDYING CRACK GROWTH IN PLASTICS COMPOSITE MATERIALS

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UNITED STATES NAVAL ORDNANCE LABORATORY, WHITE OAK, MARYLAND

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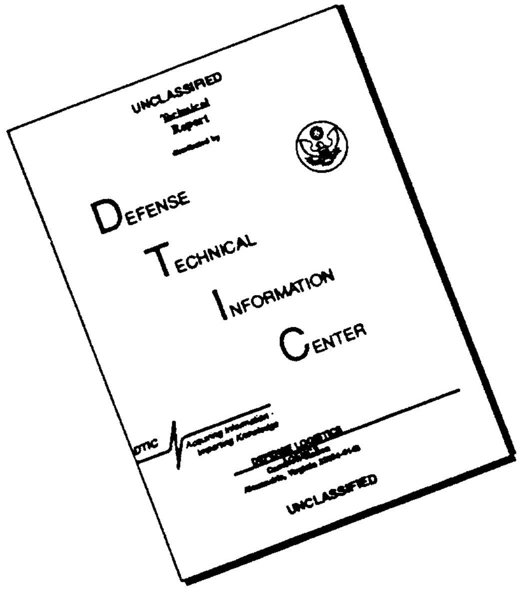

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AN AUTORADIOGRAPHIC TECHNIQUE FOR STUDYING CRACK GROWTH IN PLASTICS COMPOSITE MATERIALS

Prepared by:
Marlin A. Kinna

ABSTRACT: Preliminary work has been conducted on segments of NOL Rings to study crack propagation characteristics in fiber reinforced plastics composite materials. Samples were submitted to a variety of fatigue loading conditions. They were then immersed in a tritiated water-ethylene glycol solution and after suitable processing, autoradiograms were prepared for analysis. Radiation emanating from the solution retained by the samples indicated that minute cracks and fissures were present in all of the specimens and that the radiation intensity varied in direct proportion to the severity of the fatigue loading conditions. When photomicroscopy was used to analyze similar specimens, cracks could be detected only for the most severe test conditions.

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AN AUTORADIOGRAPHIC TECHNIQUE FOR STUDYING CRACK GROWTH IN PLASTICS COMPOSITE MATERIALS

This report presents information pertaining to the development of a technique for studying crack growth in reinforced plastics composite materials. The technique involves immersion of specimens in a radioactive tracer solution and then processing them to produce autoradiograms of the surface being analyzed. Photographs of the autoradiograms are prepared for analysis, and provide permanent records of the results.

The information presented in this report is of an exploratory nature. The tests were conducted on a single series of ring specimens which had been subjected to multiaxial fatigue loads, and the technique has not been applied to samples submitted to other types of mechanical testing. Furthermore, analytical procedures associated with the application of radioactive materials, handling of exposed specimens, and documentation of results are still in a state of flux, and the techniques described are not to be considered final. The preliminary work reported herein has been presented as papers at the 1966 Annual Technical Conference of the Society of Plastics Industry and at the first symposium on "Radioisotope Applications in Aerospace," jointly sponsored by the Wright Patterson Air Force Flight Dynamics Laboratory and the U. S. Atomic Energy Commission (February, 1966).

The technique for applying the radioactive solution and preparing the autoradiograms was developed under contract by Philco Scientific Laboratories, Blue Bell, Pennsylvania. The work was supported by Task No. RREN-ST-204/212-5/0000 and was carried out over the period from July 1964 to July 1965.

J. A. DARE
Captain, USN
Commander

Albert Lightbody

ALBERT LIGHTBODY
By direction

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ILLUSTRATIONS

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INTRODUCTION

Much work has been done in the field of fiber reinforced plastics composites to gain an understanding of the relationships between fabrication parameters, inherent mechanical properties, and crack formation and propagation. The interaction between glass fibers and resin matrices has been studied by several investigators. Erickson, et al (ref. (a)) working in this field, have found indications that factors exist at the glass-resin interface which could lead to changes in the resin-hardener ratio. The role played by glass surface finishing materials has also been widely studied. Outwater, et al (ref. (b)) for example, have shown that the surface shear strength of certain finishing materials is enhanced by boiling the finished roving for short periods of time. Working in the same area of research, Bascom (ref. (c)) found that epoxy monomers and resin liquids exhibit high contact angles on E-glass filament coated with siloxane type finishing materials. Subsequently, he showed that the number of microvoids in composite structures could be significantly decreased by reducing the contact angle to zero. In order to effectively study the relationships between fabrication parameters and the physical properties of composite structures, test methods were developed (refs. (d), (e), (f), (g), (h)) which enabled the investigator to obtain ultimate strength properties on carefully prepared samples. Although much information has been acquired as a result of these and other studies, conflicting evidence has also arisen and the actual formation and growth of minute cracks within the plastics material, when it is placed under stress, are still not clearly defined.

When plastics materials undergo mechanical failure, the failure seldom, if ever, occurs instantaneously. The time of the initial failure and the extent of the progressive degradation, however, are of prime importance. The development and progression of minute cracks are of particular interest when the structure is exposed to water or water vapor, since water is adsorbed on and interacts with the glass fibers exposed in these microscopic fissures. When the composite structure is placed under stress, unpredictable changes, influenced by the volume of absorbed water and the length of time it has been present, occur in the mechanical properties of the material. An analytical procedure is thus required which can be used to define crack initiation and propagation characteristics in reinforced plastics composite materials.

The Naval Ordnance Laboratory is studying a technique for evaluating crack initiation and propagation characteristics in plastics composite structures. A radioactive isotope is used to define the microscopic fissures which are formed under load or due to aging. The isotope penetrates the fissures and the radiation emitted can be observed by use of autoradiographic techniques. The location and concentration of the tracer material present in the structure can thus be determined, and the dimensions of the fissures can be estimated. Results for a series of NOL ring samples studied by using the autoradiographic technique are presented in this report.

BACKGROUND

Radioactive isotopes are widely used today in scientific, medical and industrial research (ref. (i)). One of the important applications is in their use as tracers. Since radioactive isotopes are chemically identical to stable isotopes of the same element, they can be used without interference to the process being carried out. Radioactive isotopes, when present in the amounts of 10^{-11} to 10^{-14} grams, can be detected using standard counting techniques, and when autoradiographic methods are used, even greater sensitivity may be obtained. Because of such high sensitivity of detection, radioisotopes have proven useful in numerous analytical procedures.

Not all radioisotopes are suitable for a given application, and selection of the correct tracer involves a number of considerations (ref. (j)). First, an isotope must have decay characteristics suitable for study of the problem at hand. Second, the experiment, when performed, should produce data of statistically sound significance within the required limits of interpretation. Third, due regard must be given to the necessary safety precautions and health hazards which exist in the handling of all radioactive materials. Consideration must also be given to other factors, such as the necessary equipment required and the ease of preparation or availability of the isotope.

Radiation is usually measured in one of two ways: (1) counting techniques or (2) autoradiography. Counting techniques involve the use of equipment suitable for recording the number of events of ionization or scintillations which are produced when radiation interacts with matter. Autoradiography makes use of a photographic emulsion to record radiation events such as gamma rays, X-rays, beta particles or alpha particles. In many applications, the emulsion is applied directly to the surface of the sample and intercepts and records radiation, incident to the emulsion, which is emitted by the radioactive isotope present in or on the specimen. The particular emulsion selected for use is determined by the type of radiation to be recorded, the level of radiation, and the degree of resolution required.

Since a solution of ethylene glycol and tritiated water of known specific activity was already available, tritium was selected as the tracer material to be used in the preliminary study of crack initiation and propagation in fiber reinforced plastics composite materials. Tritium is suitable since it radiates a beta particle which can be detected by autoradiographic techniques. In addition, when combined in proper proportions with water or other organic liquids, tritium replaces the hydrogen atom without changing the chemical characteristics of the liquid. The role played by water in the formation and propagation of internal cracks in fiber reinforced composite structures can thus be studied.

EXPERIMENTAL WORK

A series of NOL rings were fabricated and cured under identical conditions. After machining, they were submitted to a variety of multiaxial fatigue loads in the NOL Multiaxial Fatigue Tester shown in Figure 1. On completion of the fatigue tests, the portion of each ring which had received maximum stress (see Fig. 2) was used for the radioisotope analysis. A solution consisting of three parts ethylene glycol, two parts tritiated water and one part oxalic acid was used as the tracer material. The specific activity of the solution was one curie per milliliter.

The analytical procedure was carried out by placing NOL ring segments in an air-tight chamber and evacuating. The radioactive solution was then admitted to the chamber and the specimen was completely immersed in the liquid for a period of 72 hours or more. The sample was then removed from the chamber and the excess tracer material on the surface was quickly washed off. After mounting in a casting resin for easy handling, a section having a nominal thickness of 0.062 cm (0.025 inches) (thicknesses of 0.025 cm can be cut when required) was cut using liquid nitrogen as the coolant. The specimen was then stored in liquid nitrogen to prevent evaporation of the radioactive solution until coated with the photographic emulsion. Following application of the emulsion, the specimens were stored in a light tight container at 4°C for a predetermined period of time. The emulsion was then processed photographically to obtain autoradiograms, and photographs of the autoradiograms were then prepared for study. The procedure is summarized in Figure 3.

Control samples for the radioisotope analysis technique were prepared by exposing specimens in the manner described to ordinary water. Samples were prepared for each of the fatigue test conditions used.

In addition to the samples analyzed after multiaxial fatigue testing, segments of an NOL ring prepared with preimpregnated roving were analyzed. The samples were 0.5 cm long, and after exposure to the radioactive solution autoradiograms were prepared and photographed.

RESULTS AND DISCUSSION

Figure 4 shows the cross-section of an NOL ring segment which received no mechanical testing prior to immersion in the tritiated water solution. Radioactive emission is indicated by the grey and black areas present in the structure. Each such area represents a void or fissure end which is capable of retaining radioactive liquid. The largest discontinuity is approximately 6.5×10^{-3} square millimeters in area. Many are visible, however, which are only 1.5×10^{-3} square millimeters or less in area. The diagonal lines on this and other autoradiograms are machining marks.

Autoradiograms of NOL ring sections which were exposed to the radioactive solution after more extensive test conditioning are shown in Figures 5 and 6. The tracer solution has penetrated at least one lateral crack in the surface shown in Figure 5. Crack formation is also indicated by the presence of tracer material at the upper left side of the specimen. As in Figure 4, void areas which have filled with the tracer are sharply defined. Structural failure, sufficient to retain appreciable quantities of radioactive liquid, is readily indicated on the specimen surface shown in Figure 6. The shading and general configuration of many of the tracer-containing areas indicate that failure has progressed, under the test conditions imposed, in a radial manner from void areas which were present in the specimen prior to test. The radioactive tracer also indicates the presence of hairline cracks, probably just forming, in the central area of the specimen.

Figure 7 shows large quantities of tracer material which have been retained in the separations produced by severe test conditions. The large cracks are clearly defined, and smaller vertical and lateral fissures are also made apparent by the radiation. A sample taken from a position adjacent to the specimen in Figure 7 is shown in Figure 8. The autoradiogram was prepared after the sample had been processed with ordinary water. Examination of the autoradiogram led to the conclusion that the extensive damage indicated by the tritiated water solution could not be detected when ordinary water was used. Similar analysis of samples exposed to each of the other test conditions resulted in the same conclusion.

Autoradiograms of a specimen from a ring fabricated with pre-impregnated roving are shown in Figures 9 and 10. After immersion in the tritiated water solution, both ends were coated with photographic emulsion. Examination of the resulting mirror-image autoradiograms led to the conclusion that many of the voids present on the surface were indeed fissures which progressed longitudinally through the specimen for its entire length. In addition, several of the lateral cracks indicated in Figure 9 by the presence of tracer material are continuous for the length of the test specimen and appear in the mirror image presented in Figure 10. The potential value of the radioisotope technique is thus clearly demonstrated. By exposing a composite sample to a tracer material and taking sections in an appropriate manner, the length, diameter and location of internal fissures and discontinuities can be studied. The materials characteristics thus described may then be correlated with mechanical test data and ultimately may be expected to be useful in forecasting the lifetime of plastics materials.

Figures 11 and 12 are autoradiograms which have been enlarged 400 and 1800 times respectively. Fiber ends can be observed in the void areas which are completely unattached to resinous material, and excellent definition of void configuration is obtained with the use of the radioactive tracer solution, in spite of the high magnifications. Enlargements of the area around the fissure ends may provide detailed information, in subsequent studies, on the nature and origin of crack initiation.

Five specimens were taken from each sample after immersion in the tritiated water solution and were examined to evaluate the reproducibility of the test method. Comparison of the resulting autoradiograms indicated that for each of the test levels used, a distinct failure pattern was obtained. Approximately the same number of tracer-filled fissures (or bubble-like voids) appeared in all the specimens at each test level, while distinct differences, such as lateral cracks and fissures of substantial size, could be noted for the samples tested at different conditions. All observations indicated that tracer analysis offered a reliable technique for the study of internal degradation in fiber reinforced plastics materials.

Three samples which had been subjected to multiaxial fatigue loads were studied using photomicroscopy techniques. Specimens were cut, mounted and polished, and a microscopic examination of the cross-sectional surface was made. The sample which had received 5000 cycles of test at 100 cycles per minute showed a large crack across the width dimension near the center of the cross section (Fig. 13). Several smaller cracks were also forming at other locations within the structure. The remaining two samples, which had been tested at less severe conditions, showed no indication of fissures. Autoradiographic techniques, however, showed that minute cracks were indeed present in samples after fatigue testing at the less severe conditions.

The experimental techniques employed in this work are preliminary, and it is expected that more definitive data will be obtained with refinement of the method described herein. Use of tritium limited this work to an investigation of surface phenomena; other isotopes may be used in the future, however, which will permit the investigator to study internal phenomena as well. Materials which should be considered for future work are the radioactive isotopes of sodium, phosphorus, silver, etc. Since these isotopes radiate at different levels of energy, specific analytical procedures will be required for each. The variety of available materials, however, is expected to be more than sufficient to provide complete information on crack propagation characteristics in plastics composites.

The potential value of radioactive isotopes for the study of crack growth and propagation cannot be overestimated. The methods and materials used in the work discussed herein provided good resolution and clarity in the autoradiograms obtained. Special photographic emulsions are now available, however, which offer even greater resolution, and it is expected that dimensions in the order of 100 Angstroms or less may be detected. Examination of tracerized materials with photomicroscopy techniques also offers promise. Careful analysis of highly magnified surfaces generally requires the services of skilled microscopists. By photographing autoradiograms, on the other hand, surface discontinuities can be easily identified by the technician even at high magnifications. The value of isotopes will be realized to the fullest extent, however, when techniques have been developed to study the interior of plastics composite materials. By using isotopes which radiate at sufficient levels of energy, crack density profiles may be obtained which will indicate the length, size and location of discontinuities in specimens after controlled mechanical and environmental conditioning. Ultimately, techniques can be devised which will be essentially of a nondestructive nature, permitting samples to be alternately conditioned and evaluated. Valuable information on the progressive nature of failure in plastics composite materials can thus be documented.

CONCLUSIONS

The information reported herein demonstrates the potential value of radioactive tracer techniques in the study of fiber reinforced plastics composite materials. It has been shown that tracers are capable of indicating the presence of small internally situated cracks and fissures, and that the growth of such discontinuities may be followed and related to the fabrication and/or test conditions imposed upon the structure. Studies carried out on replicate samples have further indicated that the analytical procedure used herein is sufficiently reliable to produce statistically significant data.

RECOMMENDATIONS

It is recommended that additional studies be carried out leading to the development of a reliable technique for investigation of crack growth in plastics composite materials. The use of tracer isotopes other than tritium should be considered, as should the use of organic carrier materials which can be polymerized after sample exposure. On completion of these phases of study, use of the finalized procedure is recommended in all areas where basic information is required on the nature of internal changes in composite materials as a function of load and/or storage conditions.

ACKNOWLEDGMENT

The author wishes to thank Dr. Soo San Choi of the Philco Scientific Laboratories, Blue Bell, Pennsylvania, for his work leading to the development and application of autoradiographic techniques suitable for use in this study. Thanks are also extended to Dr. R. H. Cornish of the IIT Research Institute for his cooperation in the performance of the microscopic studies discussed in this report.

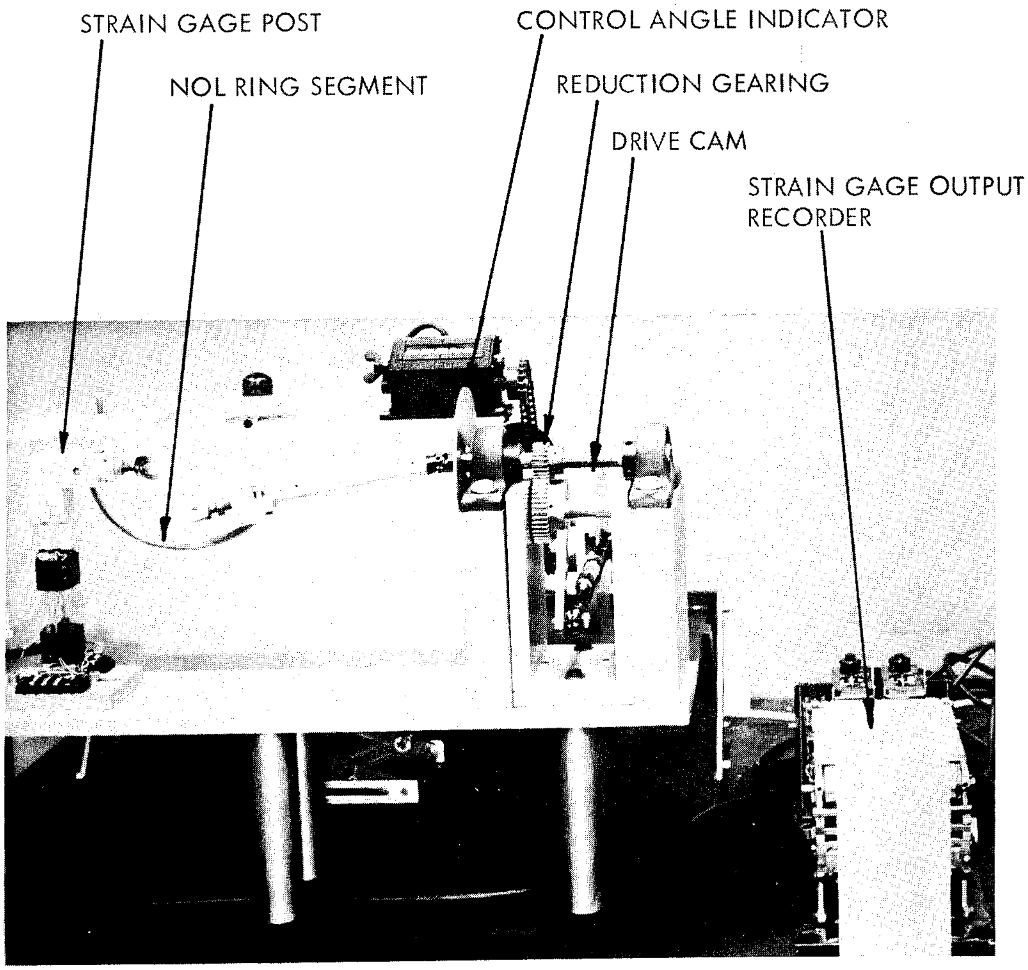


FIG. 1 NOL MULTIAXIAL FATIGUE TEST DEVICE

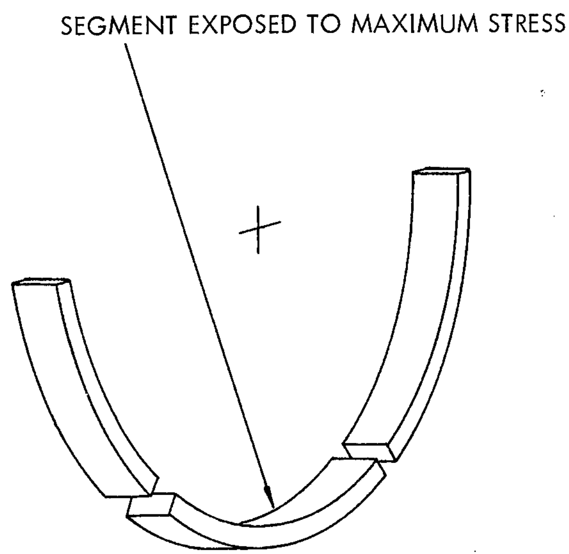
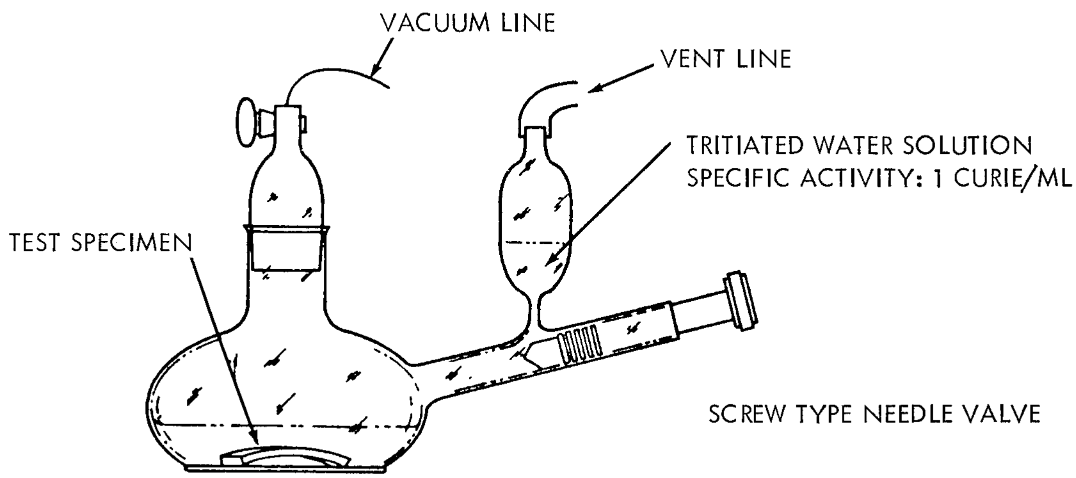


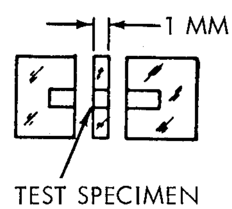
FIG. 2 SCHEMATIC REPRESENTATION OF NOL RING TEST SPECIMEN

STEP 1. IMMERSE SECTION OF FATIGUE TEST SPECIMEN IN RADIOACTIVE TRACER SOLUTION

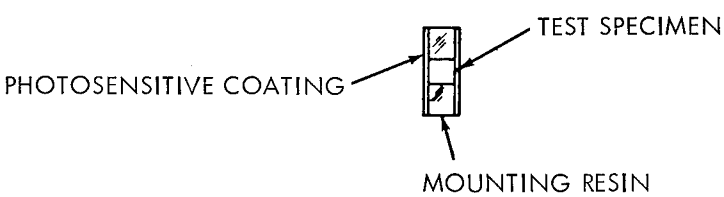


STEP 2. REMOVE AND WASH SPECIMEN

STEP 3. MOUNT SPECIMEN IN CASTING RESIN AND CUT SPECIMEN FOR ANALYSIS



STEP 4. APPLY PHOTOSENSITIVE COATING AND STORE FOR EXPOSURE



STEP 5. PHOTOGRAPH AUTORADIOGRAM FOR PERMANENT RECORD

FIG. 3 PROCEDURE FOR RADIOACTIVE TRACER ANALYSIS OF NOL RING SAMPLE

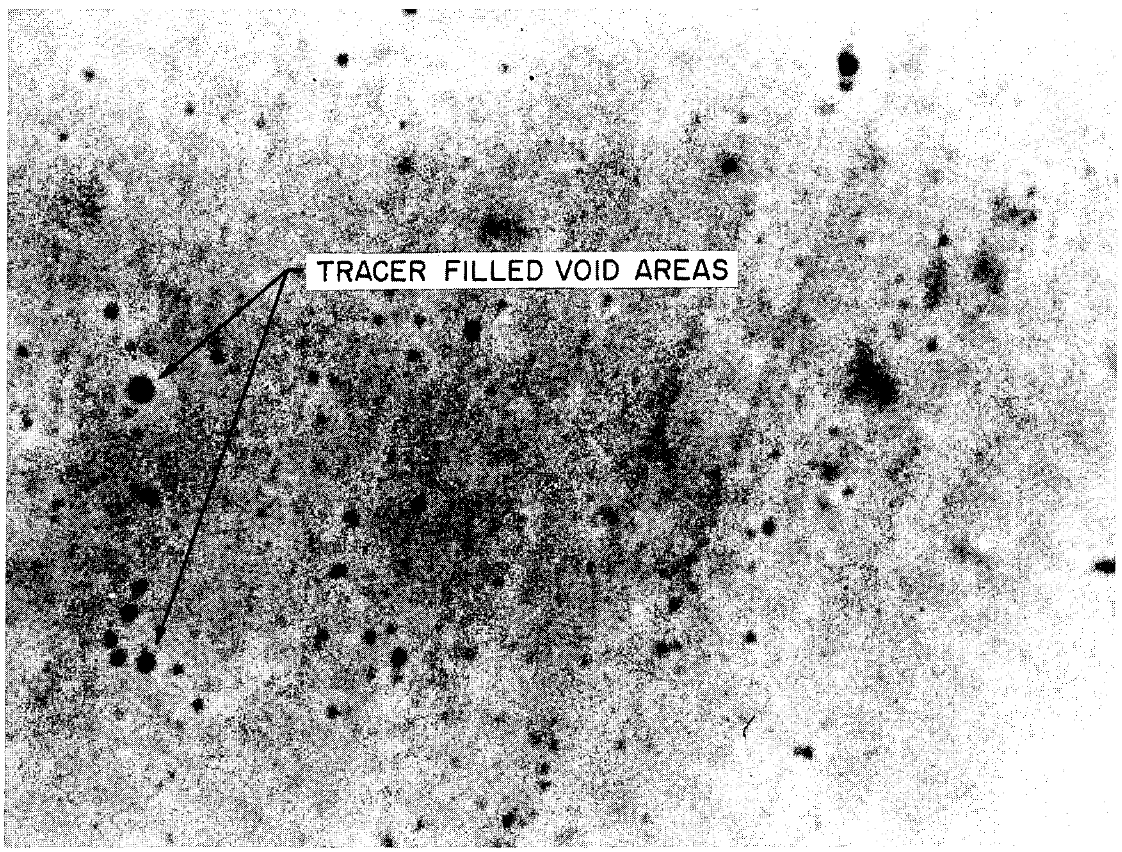


FIG. 4 AUTORADIOGRAM OF NOL RING SEGMENT (NO FATIGUE TEST CONDITIONING)
(35X)

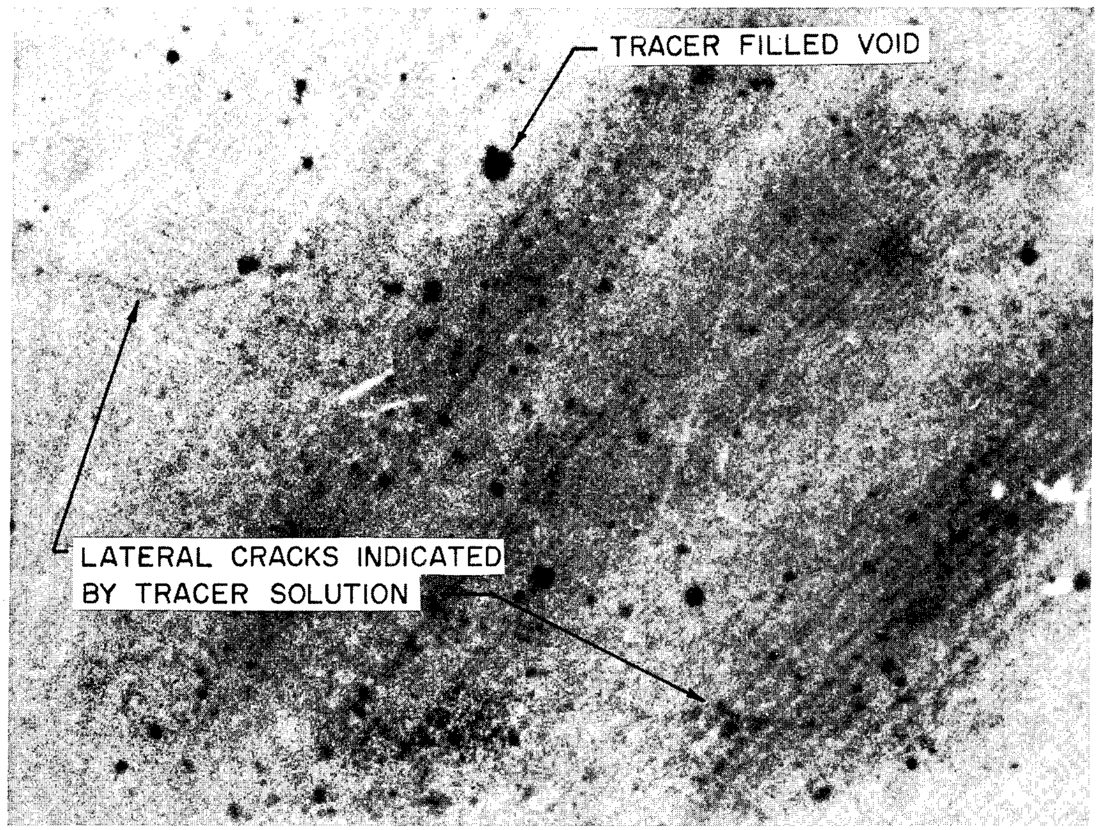


FIG. 5 AUTORADIOGRAM OF AN NOL RING SEGMENT AFTER 500 CYCLES OF FATIGUE TESTING AT 10CPM (35X)

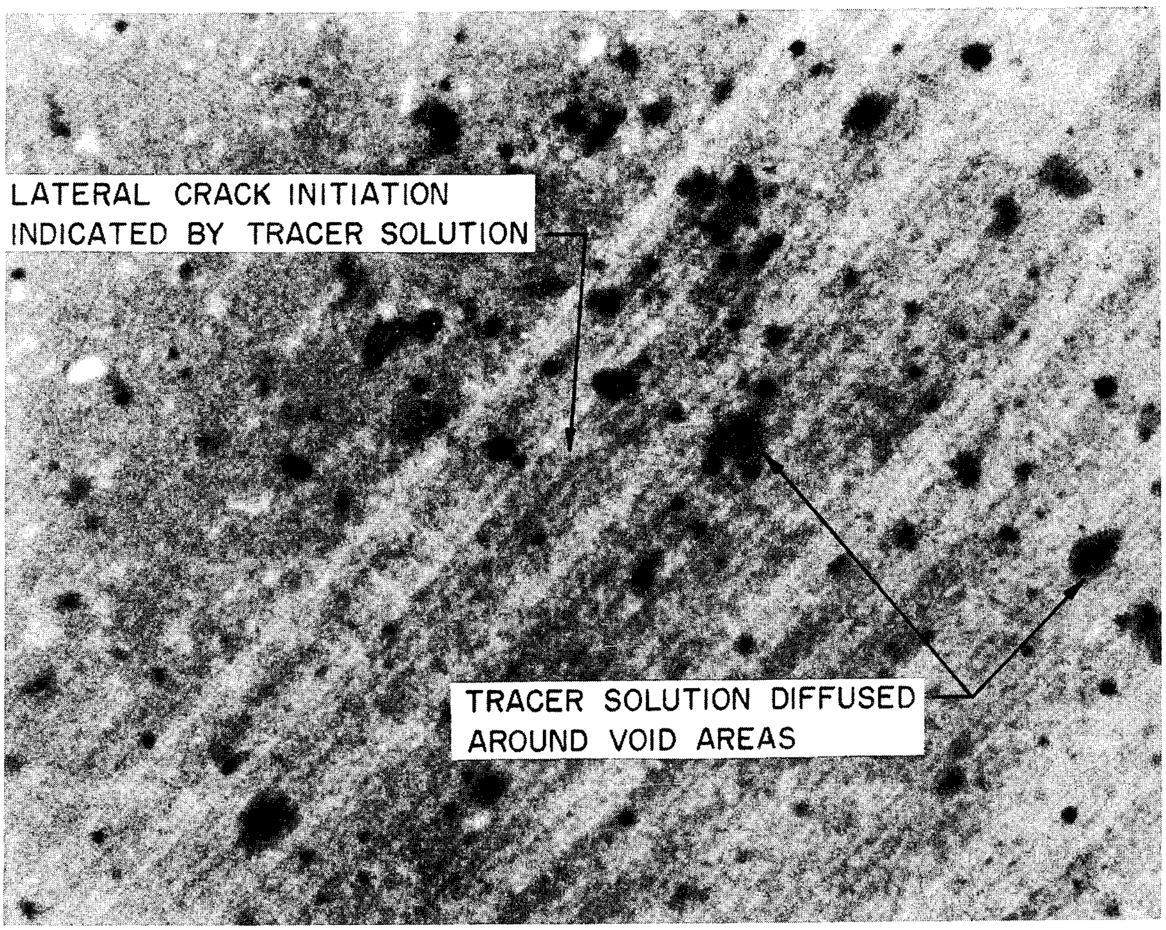


FIG. 6 AUTORADIOGRAM OF NOL RING SEGMENT AFTER 500 CYCLES OF FATIGUE TESTING AT 100 CPM (35 X)

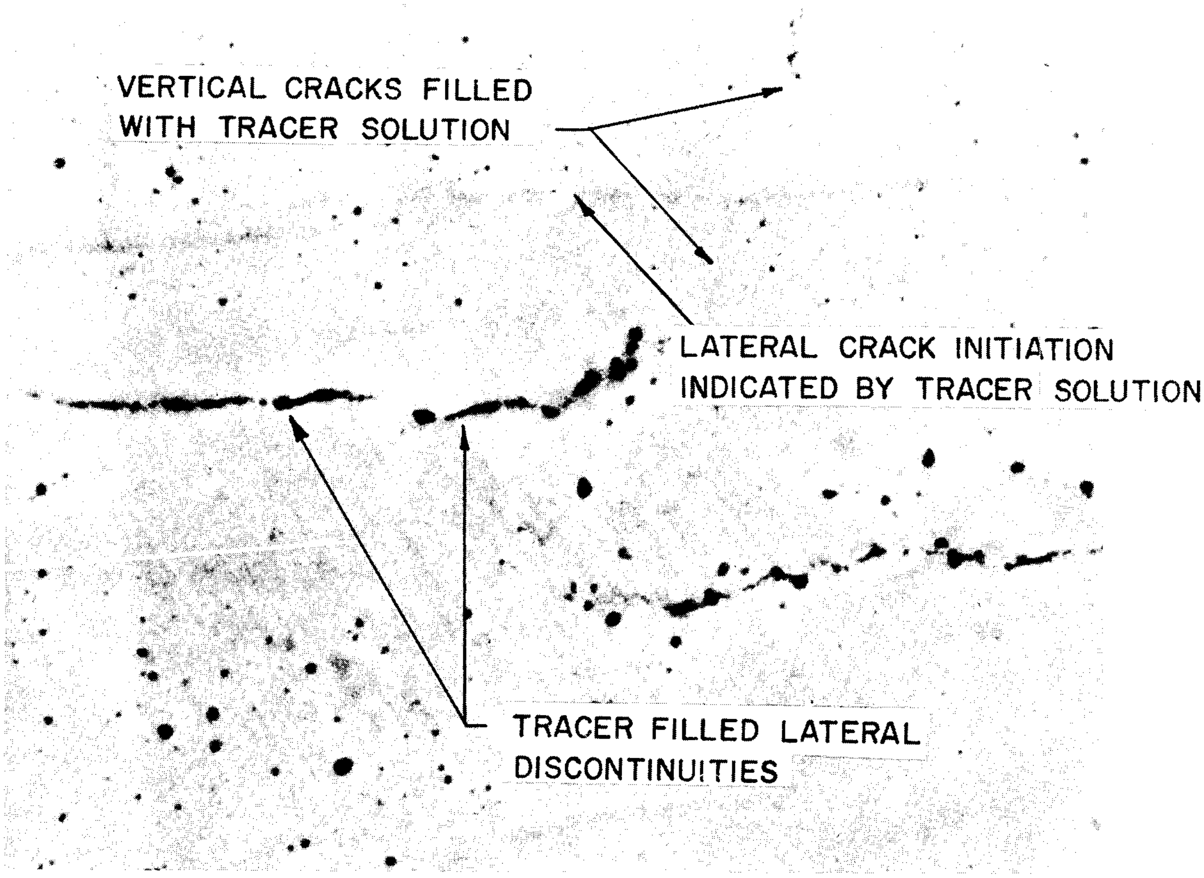


FIG. 7 AUTORADIOGRAM OF NOL RING SEGMENT AFTER 5000 CYCLES OF FATIGUE TESTING AT 100 CPM (35X)

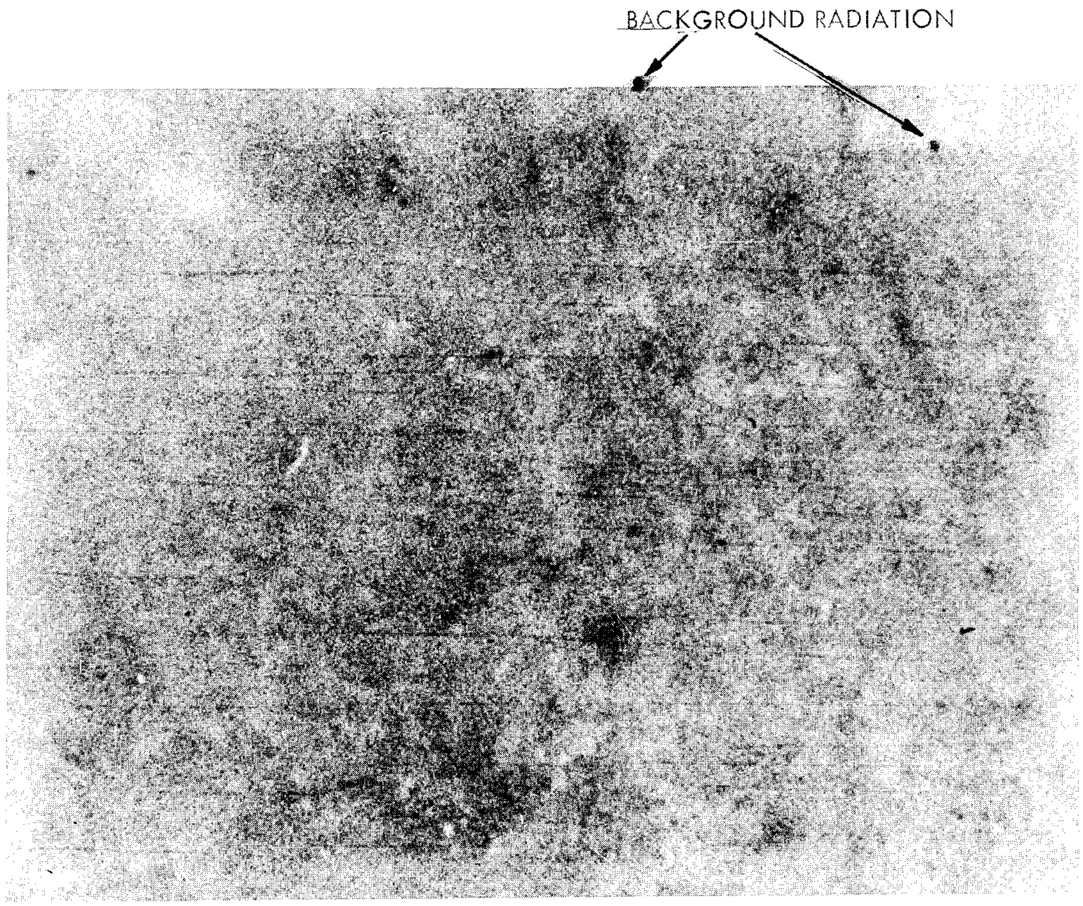


FIG. 8 AUTORADIOGRAM OF NOL RING SEGMENT AFTER 5000 CYCLES OF FATIGUE TESTING AT 100 CPM (ORDINARY WATER)(35 X)

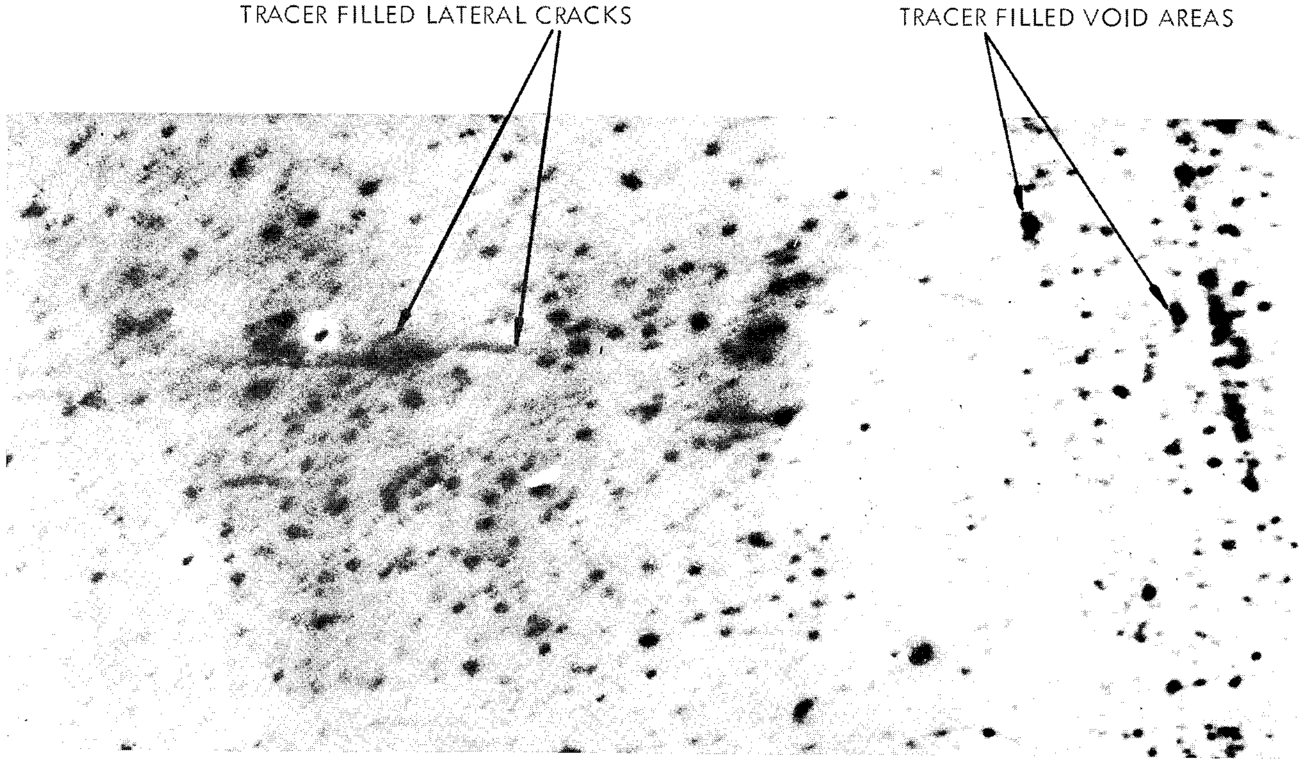


FIG. 9 AUTORADIOGRAM OF NOL RING FABRICATED WITH PREIMPREGNATED ROVING (35 X)

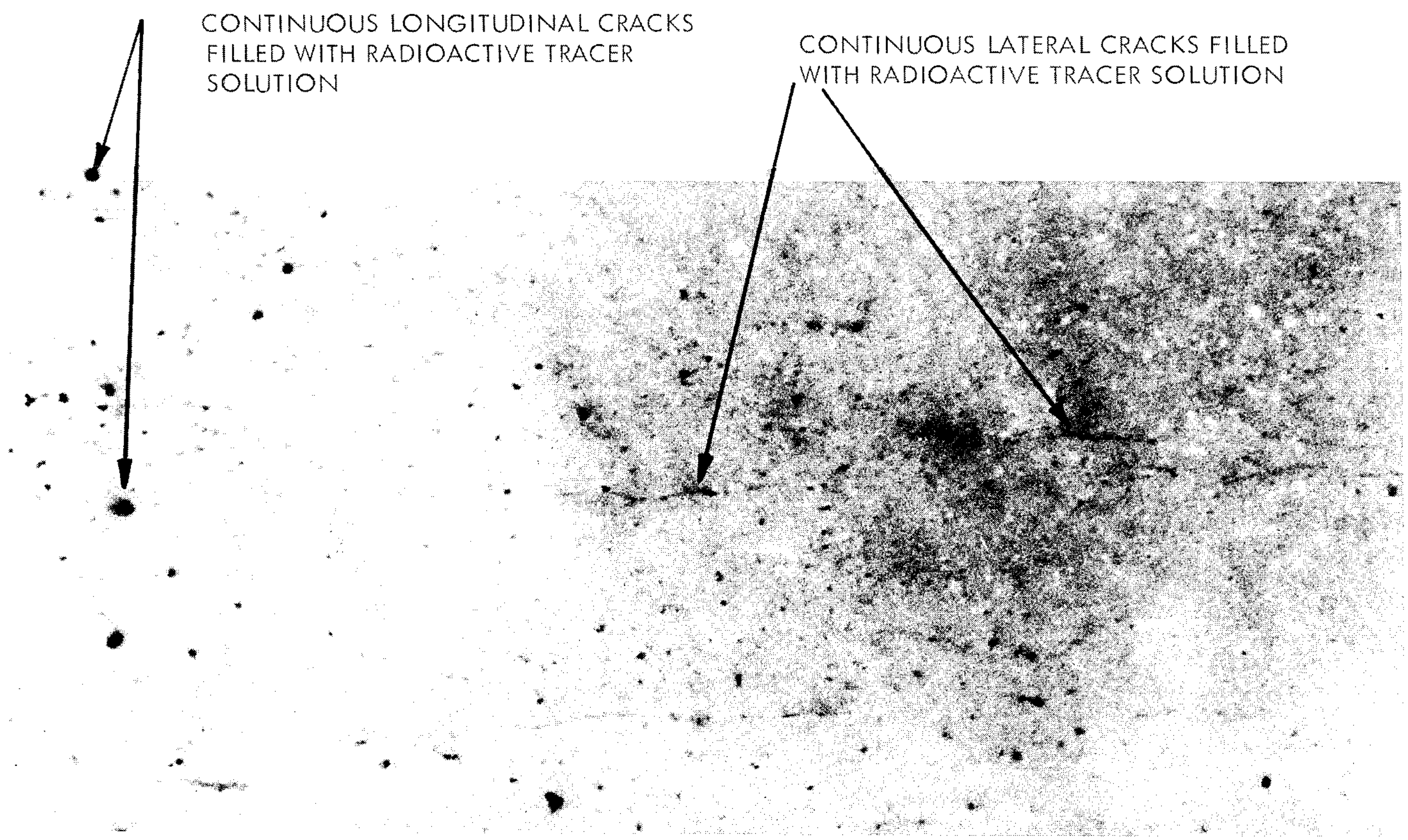


FIG. 10 MIRROR - IMAGE AUTORADIOGRAM OF NOL RING FABRICATED WITH PRE-IMPREGNATED ROVING (35 X)

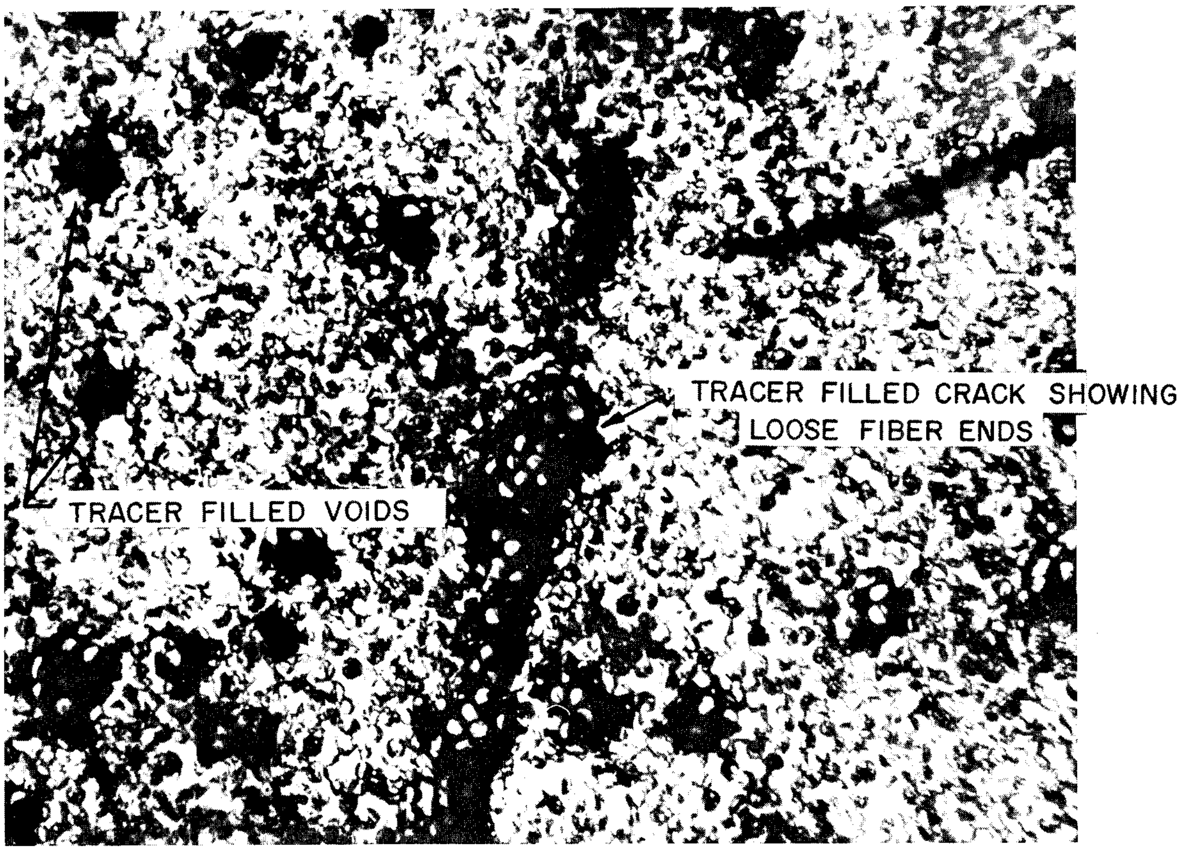


FIG. 11 PHOTOMICROGRAPH OF AUTORADIOGRAMMED NOL RING CROSS-SECTION (400 X)

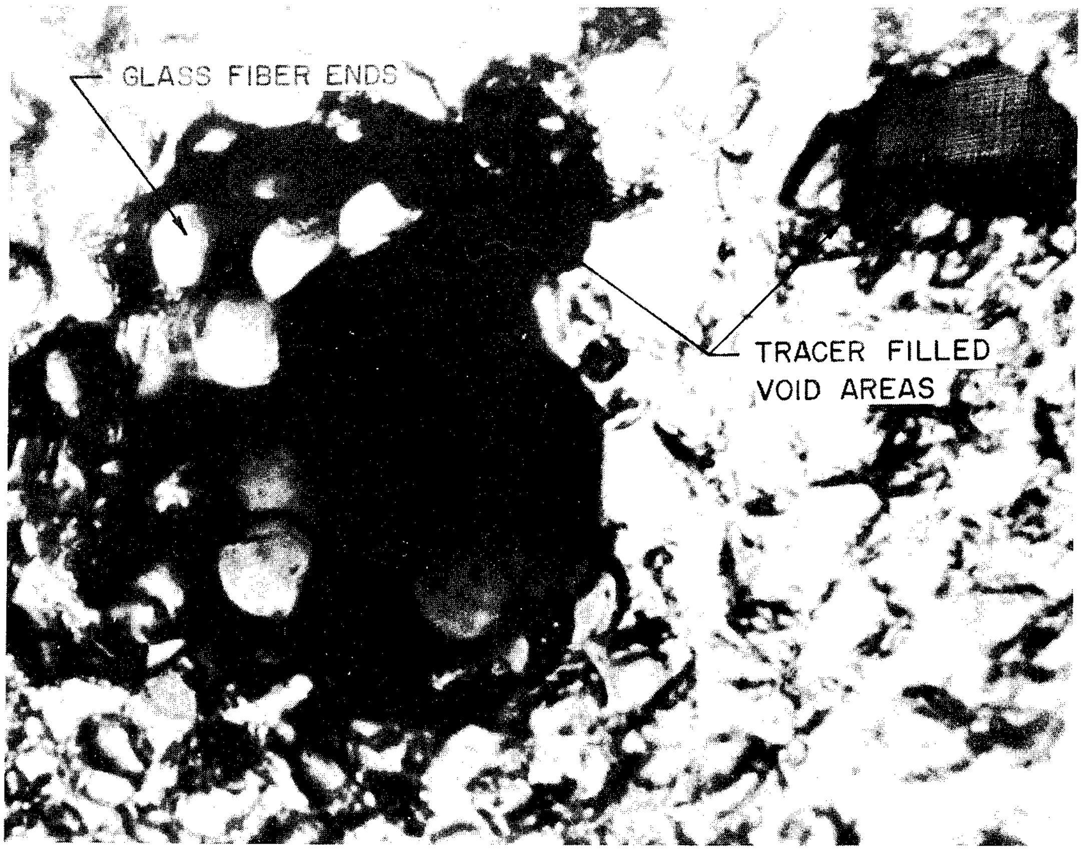


FIG. 12 PHOTOMICROGRAPH OF AUTORADIOGRAMMED NOL RING CROSS-SECTION (1800 X)

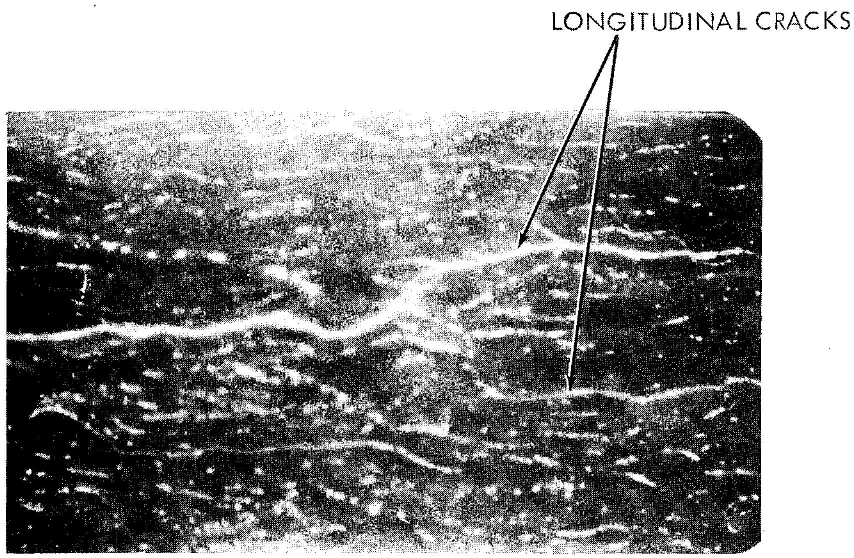


FIG. 13 PHOTOMICROGRAPH OF NOL RING SEGMENT AFTER 5000 CYCLES OF FATIGUE TESTING AT 100 CPM (35 X)

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Preliminary work has been conducted on segments of NOL Rings to study crack propagation characteristics in fiber reinforced plastics composite materials. Samples were submitted to a variety of fatigue loading conditions. They were then immersed in a tritiated water-ethylene glycol solution and after suitable processing, autoradiograms were prepared for analysis. Radiation emanating from the solution retained by the samples indicated that minute cracks and fissures were present in all of the specimens and that the radiation intensity varied in direct proportion to the severity of the fatigue loading conditions. When photomicroscopy was used to analyze similar specimens, cracks could be detected only for the most severe test conditions.

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